HYDROGEN GENERATION FOR FUEL CELLS

REPORT No. 3

THIRD
QUARTERLY REPORT
FOR THE PERIOD
1 NOVEMBER 1962 – 31 JANUARY 1963

U. S. ARMY ELECTRONICS
RESEARCH AND DEVELOPMENT LABORATORY
FORT MONMOUTH, NEW JERSEY

CONTRACT NO. DA 36-039 SC-89077

Task No. 3A99-09-002-03

ENGELHARD
INDUSTRIES, INC.

PROCESS EQUIPMENT DIVISION
NEWARK, N.J.

HYDROGEN GENERATION

FOR FUEL CELLS

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U. S. ARMY ELECTRONICS

RESEARCH AND DEVELOPMENT LABORATORY

Object: To conduct investigations on the conversion of primary fuels to hydrogen for use as a fuel in primary fuel cell batteries.

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I. PURPOSE

The work undertaken in this contract consists of investigations to develop a method of generating hydrogen from primary fuels in such a manner that the generation of the hydrogen and its separation from other reaction products can be accomplished easily in the field on a portable basis.

An experimental generator is to be designed and fabricated with the necessary reactants to produce 0.25 pounds of hydrogen distributed over a 12-hour period. The device, including fuel for 12 hours' operation, is not to exceed 15 pounds in weight or a 2-cubic foot volume.

Ammonia and hydrocarbons are to be investigated with an attempt to secure fuel cell operation at a primary fuel cost of approximately 20 mils/KWH.

The experimental generator must be easily adaptable to integration with a 200 watt, 28 volt hydrogen-oxygen fuel cell battery.

II. ABSTRACT

Test operations were continued during the third quarter with portable hydrogen generators utilizing ammonia as the fuel. Continued investigations have shown that the design requirement of 4 SCFH of hydrogen can be produced utilizing residual hydrogen to supply the process energy requirements. Repeated start ups and shutdowns have been accomplished. It is projected that a complete assembly incorporating a slightly modified version of these generators will meet the over-all volume and weight specifications.

Tests have confirmed that the waste stack gas could be utilized to provide the heat of vaporization to the ammonia container to maintain the ammonia vapor at a pressure sufficient to operate the generator. Refinement of the heat exchanger and controls still has to be engineered.

With a view to equipment simplification through elimination of a second feed stream suitable mixtures of water and JP-4 have been emulsified by simple shaking with an emulsifier.

III. PUBLICATIONS, LECTURES, REPORTS AND CONFERENCES

Conferences were held on November 15, 1962, at the Research and Development Division and the Process Equipment Division of Engelhard Industries, Inc., attended by Lt. F. R. Franke and Technical personnel of the contractor. The development work of the second quarter and the status of the present efforts were discussed.

A conference was held on January 24, 1963, at the Process Equipment Division of Engelhard Industries, Inc., attended by Dr. H. F. Hunger and Lt. F. R. Franke and Technical personnel of the contractor. The work completed at this time was discussed.

IV. FACTUAL DATA

A. Ammonia as a Fuel for Hydrogen Generators

1. Experimental Apparatus

The test generators and associated equipment were arranged as shown in the schematic flow diagram - Typical Test Setup, Figure 1. For the tests to confirm the feasibility of the stack gas to warm the ammonia supply, the equipment was arranged as shown in the schematic flow diagram - Test Setup, Figure 2.

2. Generator Performance

Operation of test reactor PT-3, which went on stream during the second quarter, was continued to secure life and performance data. The apparatus was arranged as shown in Figure 1. Operation was on a daily basis, 8 hour on-16 hour off cycle, with complete cooling in the off period. The generator failed during the 21st cycle. A leak of process gas into the pure hydrogen stream developed due to thermal expansion stresses.

During the period of testing PT-3, a modified test generator for producing H_2 from ammonia designated PT-5 was designed and built. This generator was designed without a heat distributor such as used in PT-3. Instead, it utilized a heavier walled combustion section to distribute the heat. The reaction zone had the same volume as previous generators but was shorter in length in an effort to minimize the over-all equipment height. Feed gas flow was parallel to stack gas flow.

For initial tests of PT-5, spherical rather than granulated catalyst was selected to minimize the problem of fines in the system. The generator developed a leak, during the second cycle, between the reactor gas and the pure product hydrogen. Operation was continued without hydrogen removal in order to check the temperature profile and compare the catalyst activities. Test data indicated

that the particular catalyst used initially was not sufficiently active to promote complete dissociation of the ammonia under the reactor conditions used. The larger mass of the combustion chamber wall increased the start up time of the generator. In this generator, the residual fuel off gas was conducted to the aspirator assembly at a higher temperature than previously. This higher temperature reduced the flow rates of both the residual gas and the aspirated air.

The generator then was disassembled and the leak (mentioned above) was found to be at the weld of a flange to the combustion chamber. Since the defect developed at the junction between two components and not in a component itself, we conclude that the materials of construction of the generator are now sufficiently rugged and that the remaining problem is one of assembly techniques. The generator was modified and reassembled, with the feed gas flow now countercurrent to the stack gas flow. The generator was operated again to check start up times and the reactor performance. The catalyst was changed to one which under comparable conditions proved to be more active.

Typical test data are shown in the Test Data for Ammonia Fuel, Table 1. Typical temperature profiles for PT-3, PT-5 parallel flow, and PT-5 countercurrent flow are shown in Figures 3, 4, and 5, respectively. The temperature distribution curves shown on Figures 3, 4 and 5, show that all points of the systems studied the temperatures were within the range required for proper operation of the generator.

With countercurrent flow, the generator was better than with parallel flow in regard to start up and operating characteristics. Nevertheless, this generator still did not compare as favorably to the design requirements as did prototype PT-3. Consequently, we constructed a new generator, designated PT-3A, in which were combined the improved mechanical features of PT-5 and the attractive operating characteristics of PT-3. This unit has so far been operated successfully through 10 cycles as shown in Table 2. During these cycles, the unit was self-sustaining and met the design requirements.

3. Ammonia Feed System

In this work period, attention has been given to the problem of feeding the ammonia vapor into the generator reliably under a variety of ambient conditions. This requires provisions to supply the heat of evaporation to the ammonia feed vessel and, furthermore, it would be desirable to incorporate a device separating the ammonia vapor from the liquid so that only vapors will enter the generator regardless of attitude.

a. Ammonia Vaporizer

During several tests on PT-5, the equipment was arranged as shown in Figure 2. The stack gas was conducted through a coil which surrounded a small ammonia container, five-inch diameter by seven inches long. The container and coil were lightly insulated.

The generator was operated at design conditions producing 4 SCFH of hydrogen on a self-sustaining basis while drawing ammonia vapor from the small container which was receiving the heat for vaporization from the waste stack gas. Temperature of the liquid in the ammonia container was maintained at slightly above room temperature.

This same test was successfully repeated several times using PT-3 generator.

b. Ammonia Separator

A teflon coated ceramic candle with a 1.4 micron porosity was mounted as shown in Figure 7. The container was filled to approximately 70% of capacity with anhydrous ammonia. With the container in a vertical position (candle at top), the outlet valve was opened and vapor discharged satisfactorily. The container was then inverted so that the candle was completely immersed in the liquid and the outlet valve opened. Ammonia liquid was discharged immediately.

In both tests, the discharge was to atmospheric pressure with a correspondingly large pressure drop across the candle. It is planned to check the scheme further and test the operation when the pressure drop across the filter is minimized. Additional testing will also be done to further explore the possibility of operating with a partially flooded candle.

B. Hydrocarbon as Fuel for Hydrogen Generation

1. Emulsifiers

For a small generator, as required by the present specifications, it might be simpler to have a single fluid feed to the generator instead of two separately metered and proportioned feeds. Investigations on hydrocarbon fuels have shown that it is possible to employ liquid hydrocarbons, such as JP-4 jet fuel, and water, as feed material to a hydrogen generator. Experiments have been carried out with emulsifiers fc the JP-4/water system in suitable ratios. A summary of various emulsifier systems which have been screened is given in Table 3. These tests were performed with at least one part emulsifier per four parts of oil. The only agitation utilized in these experiments was simple hand shaking.

A review of the screening tests indicated that both 9D-208 and Petromix 10 were successful in securing a stable emulsion. However, only 9D-208 was successful when the proportion of emulsifier was reduced. To date, 9D-208 has formed a stable emulsion (greater than 48 hours) with the JP-4/water system in proportions as low as one part emulsifier per 16 parts of oil.

2. Process

No process studies of liquid hydrocarbon fuel have been carried out during the Third Quarter with funds provided by this contract.

V. CONCLUSIONS

Several versions of an ammonia fueled hydrogen generator have been constructed, tested and operated to failure. All failures have been of a mechanical nature. By analyses of test operations and these failures, it has been possible to arrive at a mechanical and process design which appears sound and capable of being engineered into a practical unit.

Operational tests of prototype generators have continued to demonstrate the feasibility of obtaining 4 SCFH of hydrogen utilizing the residual off-gas as fuel.

The waste stack gas from the generator can be utilized, by heat exchanger, to provide the heat necessary to vaporize liquid at a rate to keep the generator operating on a self-sustaining basis.

VI. PROGRAM FOR NEXT INTERVAL

It is planned to finalize and optimize the design of the ammonia fueled portable hydrogen generator. Minimum weight, volume and start up times will be continued objectives. The operation of the aspirator and igniter system will be monitored for possible improvement. The method of heat transfer to provide the heat of vaporization to the ammonia container will be investigated further together with the necessary control system.

Work will be continued to investigate possible methods of vapor-liquid separation in the ammonia container in conjunction with heat transfer developments.

For the particular size and application of a hydrogen generator, methanol is an attractive fuel and it is intended to begin investigations of the process conditions and equipment design for a unit using methanol as the feed stock.

We expect to continue work with emulsified mixtures of water/hydrocarbon and to observe the behavior of such mixtures and the effects of emulsifier additives in a generator.

VII. IDENTIFICATION OF KEY TECHNICAL PERSONNEL AND DISTRIBUTION OF HOURS

The following technical personnel have been working on the hydrogen generator for the quarter:

	Hours
Mr. D. J. Accino	10
Mr. E. J. Emerson	266
Mr. H. H. Geissler	68
Mr. L. Kantrowitz	482
Mrs. R. Rylander	8
Technicians	1890

APPENDIX A

Tables

TABLE 1

SUMMARY OF DATA FOR AMMONIA FUEL

Portable Hydrogen Generator

Reactor Designation	Cycle No.	Product H_2 (SCFH)	Total H_2 in Prod. (SCFH)	Ammonia Feed (SCFH)	% H_2 in Fuel Gas	Mat'l. Eff. (lb H_2/lb NH_3)	Therm. Eff.* (GHV H_2/GHV NH_3)
PT-3	13	4.2	7.4	4.9	57.1	0.099	62.9
	16	4.0	7.2	4.8	57.1	0.097	61.6
	20	4.3	7.2	4.8	54.7	0.104	66.0
PT-5 (Parallel Flow)	2	1.9	6.2	4.1	68.3	0.054	34.3
PT-5 (Counter-current Flow)	2	3.8	7.8	5.2	60.6	0.085	54.0
	3	4.0	7.95	5.3	59.8	0.088	56.9
	4	3.5	6.6	4.4	58.5	0.092	58.4

* Thermal Efficiency = Gross Heating Value Product H_2/Gross Heating Value NH_3

TABLE 2

SUMMARY OF DATA FOR AMMONIA FUEL

Portable Hydrogen Generator--PT-3A

Cycle No.	Product H_2 (SCFH)	Total H_2 in Prod. (SCFH)	Ammonia Feed (SCFH)	% H_2 in Fuel Gas	Mat'l. Eff. (lb H_2/lb NH_3)	Therm. Eff.* (GHV H_2/GHV NH_3)
1	4.5	7.6	5.05	55.4	0.103	65.4
3	4.7	8.1	5.4	55.7	0.101	64.1
6	4.8	8.55	5.7	56.8	0.098	62.2
8	4.5	7.8	5.2	55.9	0.100	63.5
10	4.4	7.35	4.9	54.6	0.104	66.0

* Thermal Efficiency = Gross Heating Value Product H_2/Gross Heating Value NH_3

TABLE 3

EMULSIFIER SCREENING TESTS[1]

Emulsifier System	Commercial Source	Results
Tergitol 7	Union Carbide	Complete Separation
" 14	" "	" "
" NP-27	" "	" "
" NPX	" "	" "
Tergitol 14+ Tergitol NP-27	" "	" "
" 14+ " NPX	" "	" "
" NP-27+ " NPX	" "	" "
Triton X- 45	Rohm and Haas	" "
" X-100	" "	" "
" GR-7	" "	" "
Triton 45+) " 100+)Mixture " GR-7)	" "	Stable for Few Hours
Triton N-57	" "	Complete Separation
9D-208	" "	Stable for 100 Hours
Petromix 10	Sonneborn Corp.	Stable for 100 Hours
Petronate HL	" "	Complete Separation

(1) These emulsifiers were selected after contacting various companies and reviewing their recommendations in regard to the conditions and requirements of a successful emulsion.

APPENDIX B

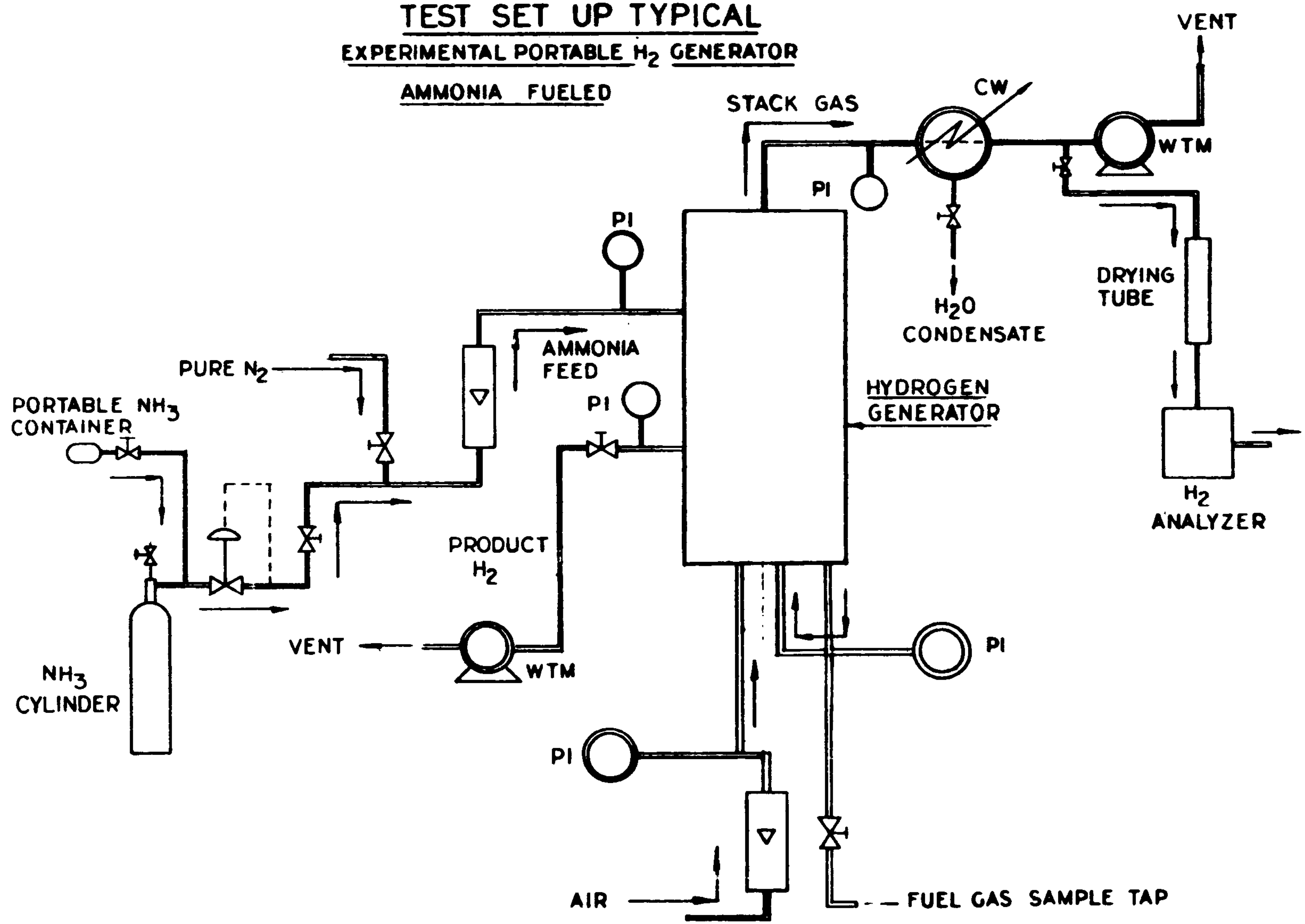
Figures

FIG. I

SCHEMATIC FLOW DIAGRAM
TEST SET UP TYPICAL

EXPERIMENTAL PORTABLE H_2 GENERATOR

AMMONIA FUELED



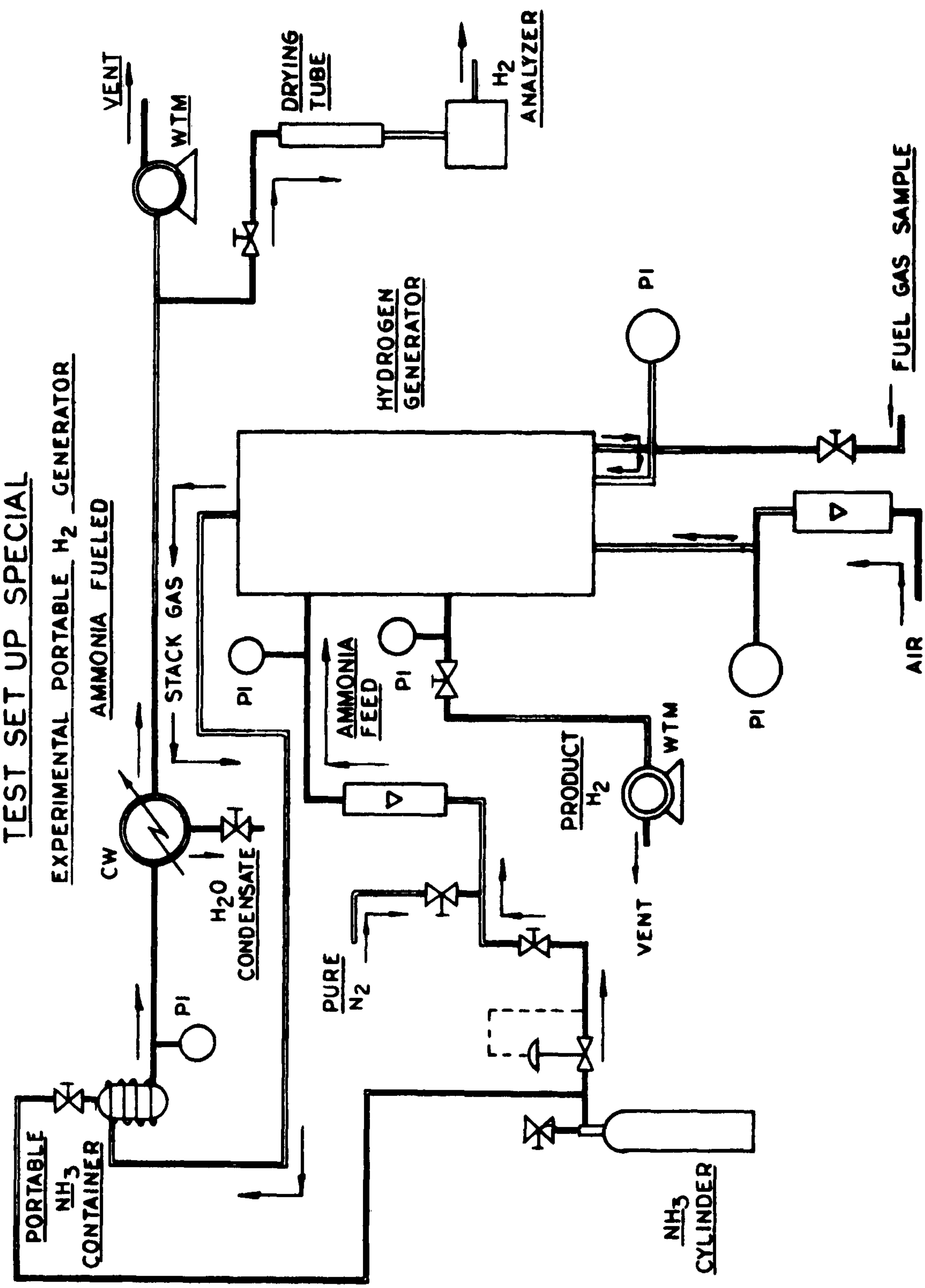


FIG. 2
SCHEMATIC FLOW DIAGRAM
TEST SET UP SPECIAL
EXPERIMENTAL PORTABLE H_2 GENERATOR
AMMONIA FUELED

FIG. 3
TEMPERATURE PROFILE
PORTABLE H_2 GENERATOR
PT-3

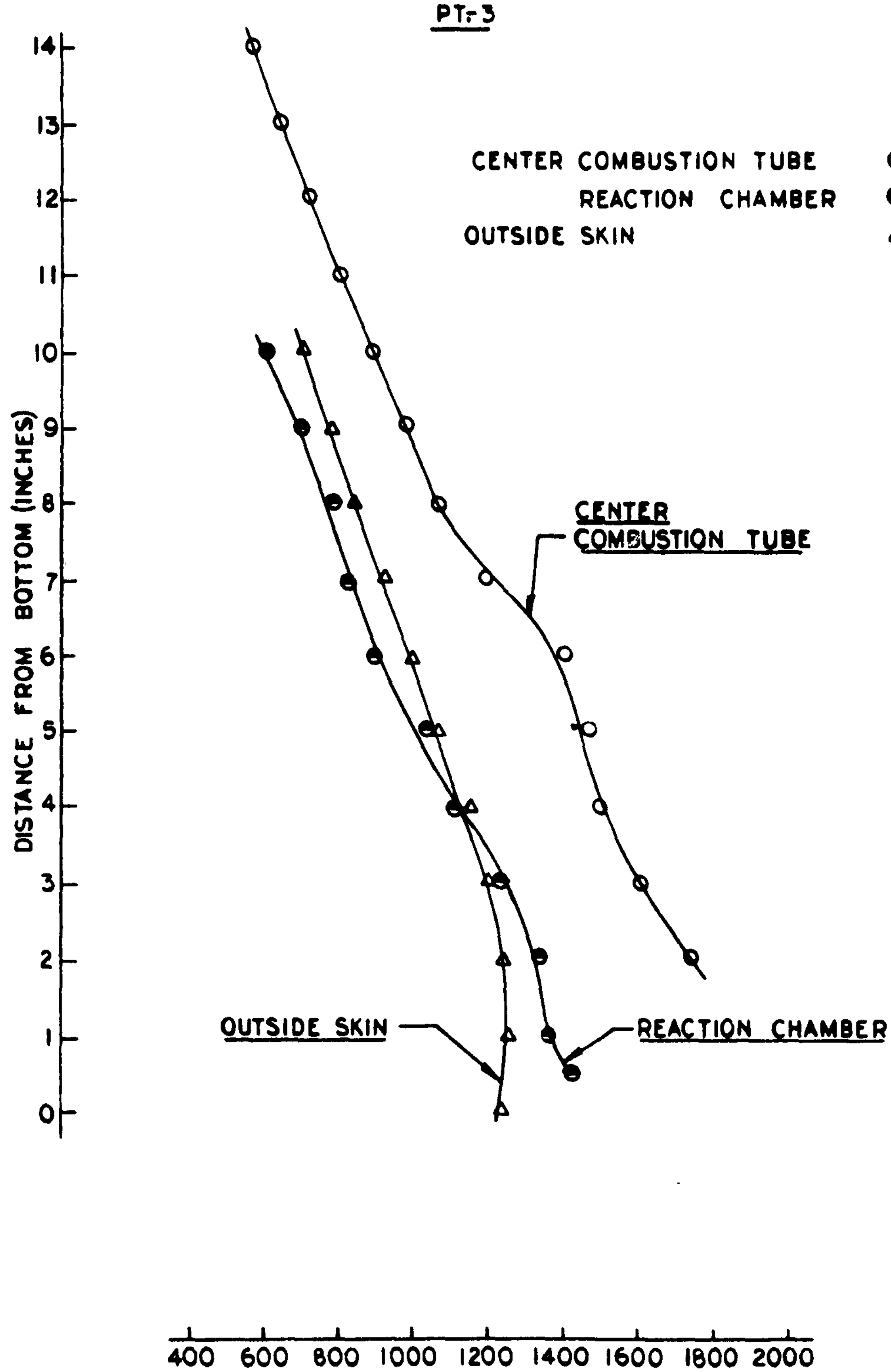


FIG. 4
TEMPERATURE PROFILE
PORTABLE H_2 GENERATOR
PT-5

PARALLEL FLOW

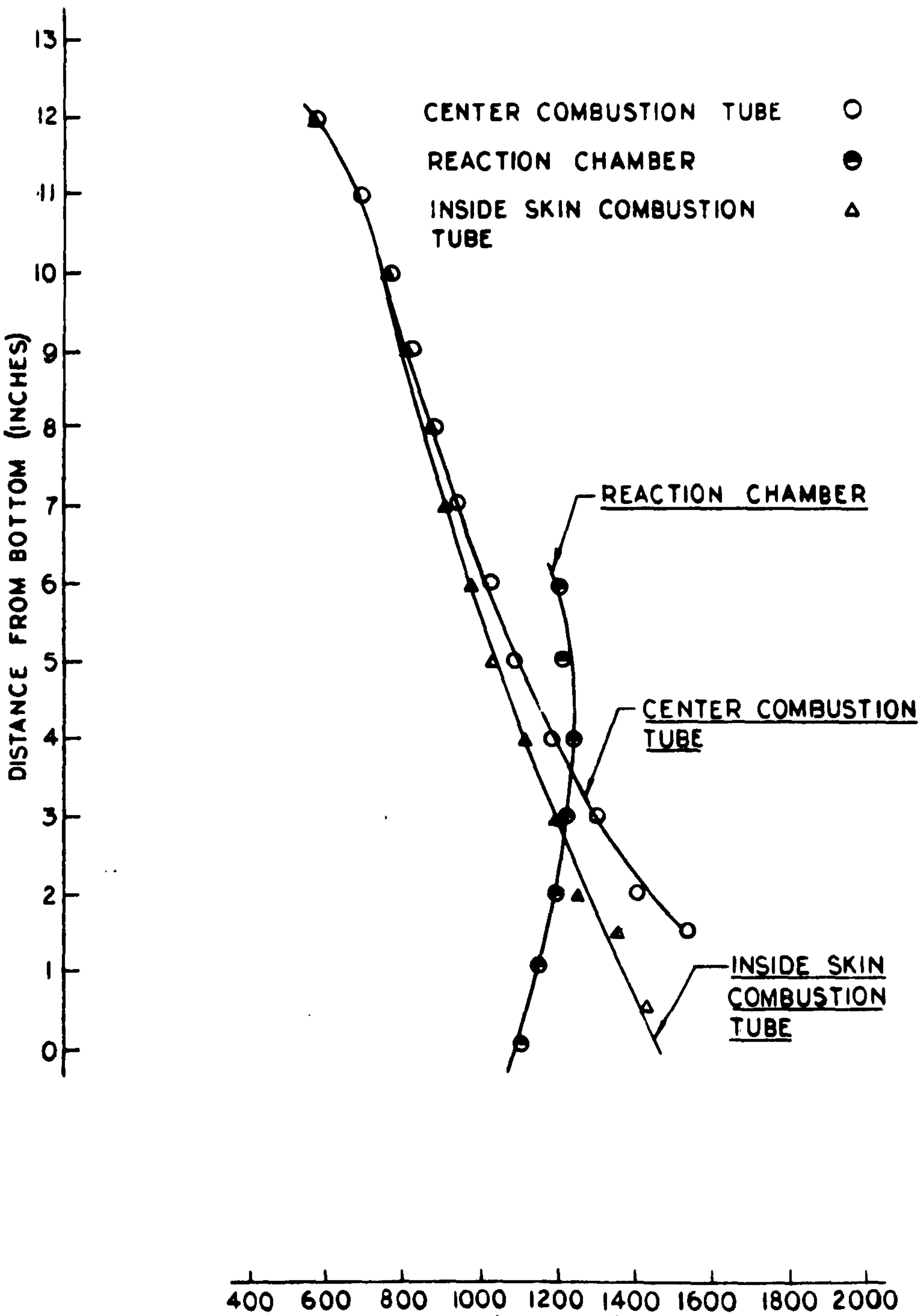


FIG. 5
TEMPERATURE PROFILE
PORTABLE H_2 GENERATOR
PT.-5
COUNTERCURRENT FLOW

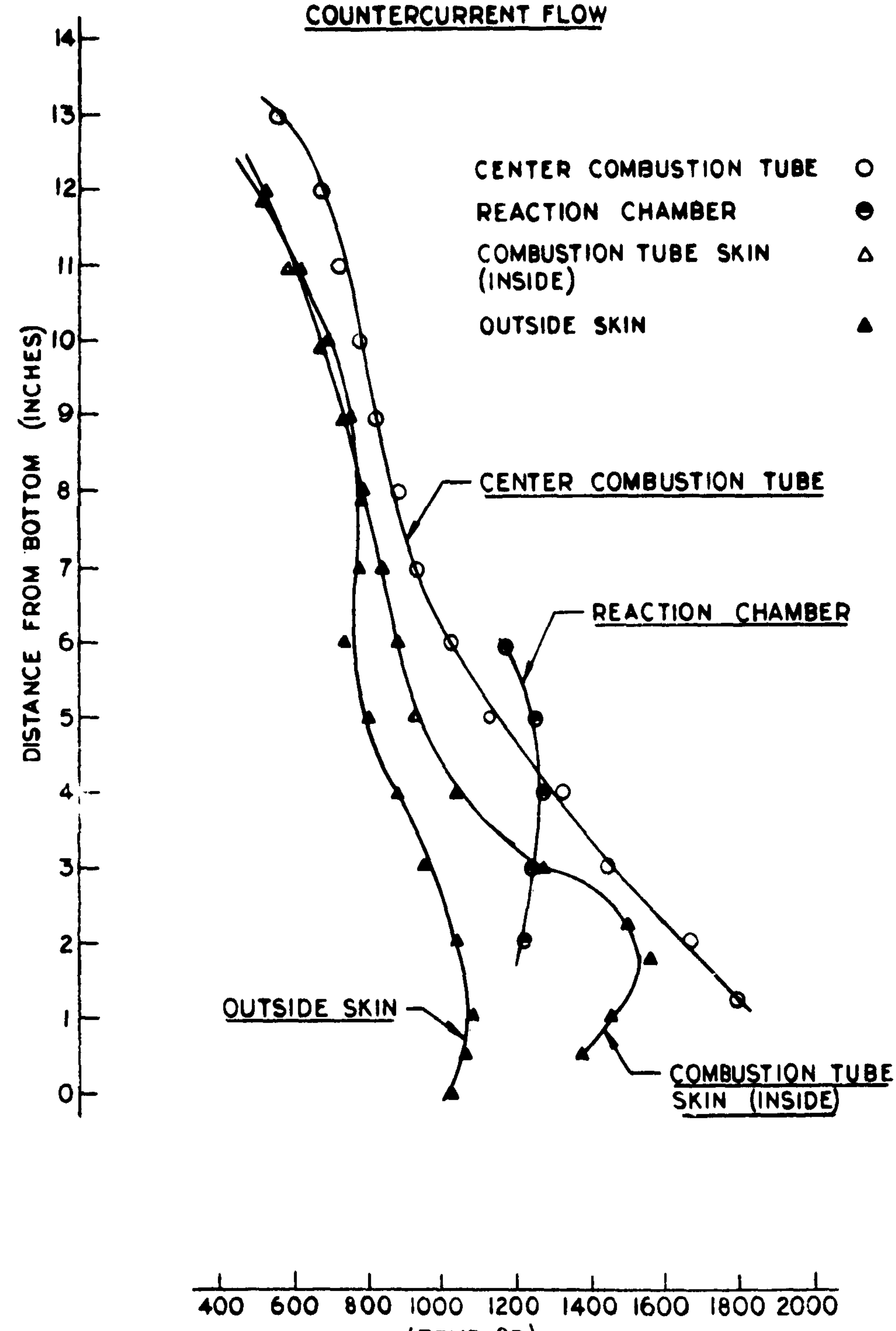


FIG. 6

TEMPERATURE PROFILE

PORTABLE H_2 GENERATOR

PT-3A

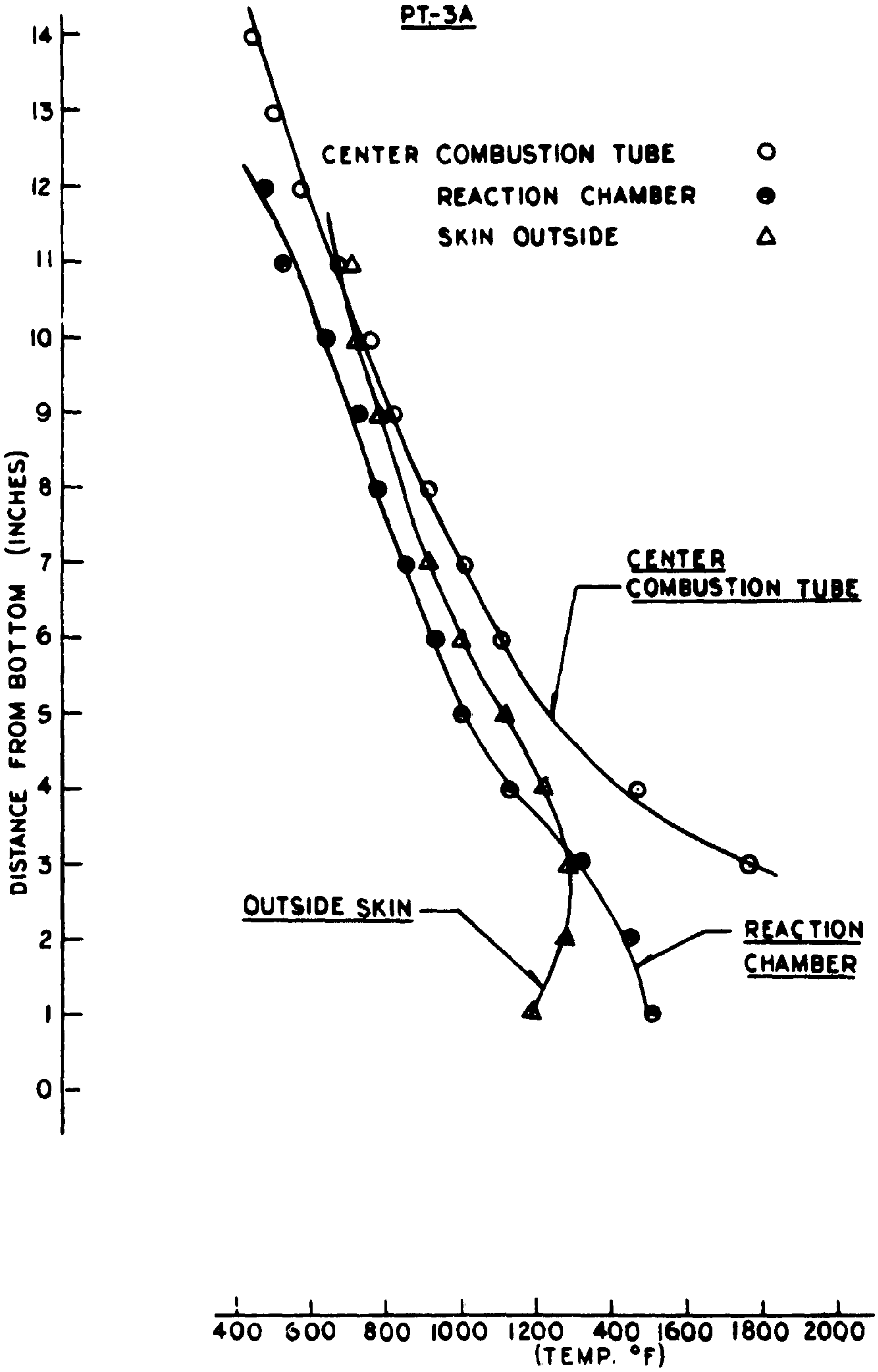
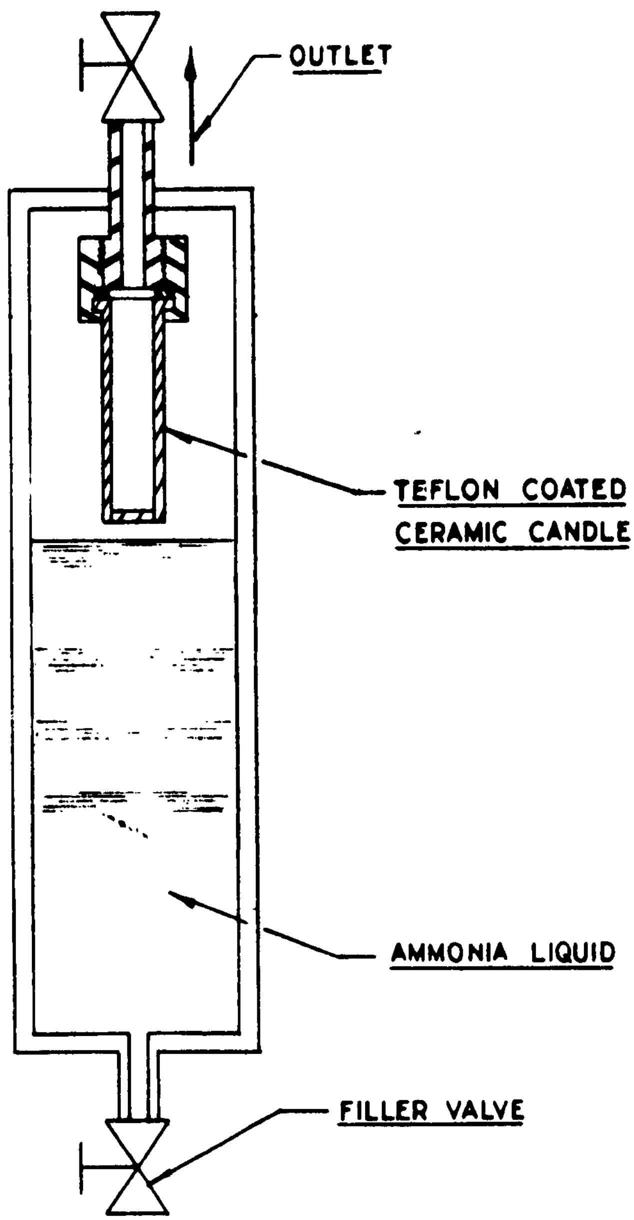


FIG. 7
AMMONIA SEPARATOR



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by E. J. Emerson, L. Kantrowitz,
H. H. Geissler. Rept. No. 3
1 November 1962 - 31 January 1963
22 p. incl. illus. tables.
(Contract DA 36-039 SC-89077)

UNCLASSIFIED REPORT

A research project is carried out to develop a method of generating hydrogen from primary fuels in such a manner that the generation of hydrogen and its separation from other reaction products can be accomplished in the field on a portable basis. Successful operations continued on a portable hydrogen generator utilizing ammonia feed at a pressure of 35 psig. The waste stack gas was utilized successfully as the source of heat of vaporization

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1. Electric power supply by fuel cells.
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